Bioresource Technology 123 (2012) 98–105

Contents lists available at SciVerse ScienceDirect

Bioresource Technology

journal homepage: www.elsevier.com/locate/biortech

Torrefaction and low temperature carbonization of oil palm fiber and eucalyptus in nitrogen and air atmospheres

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HIGHLIGHTS

- ▶ Property variation of biomass pretreated in nitrogen and air atmospheres is studied.
- ▶ An herbaceous biomass (oil palm fiber) and a wood (eucalyptus) biomass are considered.
- ▶ The reaction temperature is in the range of 250–350 °C and the duration is 1 h.
- ▶ An energy-mass co-benefit index (EMCI) is introduced to seek the optimum operation.
- ▶ EMCI simultaneously considers energy yield and solid yield.

ARTICLE INFO

Article history:
Received 21 January 2012
Received in revised form 20 June 2012
Accepted 25 July 2012
Available online 2 August 2012

Keywords:
Torrefaction
Low-temperature carbonization
Air and nitrogen
Oil palm fiber and eucalyptus
Energy and solid yields

ABSTRACT

Torrefaction is a pretreatment method for upgrading biomass as solid fuels. To provide flexible operations for effectively upgrading biomass at lower costs, the aim of this study was to investigate the properties of oil palm fiber and eucalyptus pretreated in nitrogen and air atmospheres at temperatures of 250–350 °C for 1 h. Based on energy and solid yield and introducing an energy-mass co-benefit index (EMCI), oil palm fiber pretreatment under nitrogen at 300 °C provided the solid fuel with higher energy density and less volume compared to other temperatures. Pretreatment of oil palm fiber in air resulted in the fuel with low solid and energy yields and is therefore not recommended. For eucalyptus, nitrogen and air can be employed to upgrade the biomass, and the suggested temperatures are 325 and 275 °C, respectively.

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1. Introduction

Biomass can be converted to energy or energy carriers by thermochemical and biochemical processes (Goyal et al., 2008). Thermochemical processes include combustion, gasification, liquefaction, hydrogenation and pyrolysis which can convert biomass into heat, solid, liquid and gaseous fuels (Yaman, 2004; Goyal et al., 2008). However, raw biomass can have characteristics that may lower processing efficiency. For example, the oxygen content in raw biomass is around 30%; its calorific value is thus merely about 50% that of conventional fossil fuels (Swithenbank et al., 2011). Low bulk and energy densities as well as the high moisture content of raw biomass also decrease energy efficiency (Werther et al., 2000) and increase storage and transportation costs. Torrefaction can effectively improve the properties of biomass and lead to a higher conversion efficiency (Uslu et al., 2008).

Torrefaction is a thermal pretreatment process for producing solid fuel in which raw biomass is heated in a non-oxidizing atmosphere in the temperature range of 200–300 °C. The main advantages of torrefaction include amplification of the energy density of biomass (Arias et al., 2008; Deng et al., 2009; Almeida et al., 2010; Chen et al., 2011a, 2012; Phanphanich and Mani, 2011; Rousset et al., 2011; van der Stelt et al., 2011; Li et al., 2012), improvement of its grindability (Arias et al., 2008; Deng et al., 2009; Bridgeman et al., 2010; Repellin et al., 2010; Phanphanich and Mani, 2011) and conversion of a hygroscopic biomass into a hydrophobic material (Acharjee et al., 2011; Li et al., 2012). Utilizing torrefied biomass is more efficient than consuming raw biomass in combustion (Bridgeman et al., 2008; Pimchuai et al., 2010; Rousset et al., 2011), pyrolysis (Wannapeera et al., 2011) and gasification (Couhert et al., 2006; Prins et al., 2006a; Deng

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http://dx.doi.org/10.1016/j.biortech.2012.07.096

et al., 2009). Torrefied biomass can also be pelletized easier, thereby reducing storage and transportation costs (Uslu et al., 2008; Bergman et al., 2005). Carbonization is another thermal process by which biomass is treated in an inert atmosphere at temperatures of 277–677 °C (Maschio et al., 1992); heating rate and operating pressure are limitations in this process.

The use of inert atmospheres for torrefaction and carbonization contributes to the cost of the processes. A reduction in cost could potentially be achieved if the processes could be carried out in air. To evaluate the potential of upgrading biomass using air as a carrier gas, a herbaceous and a woody biomass were carbonized in nitrogen and air to analyze the variations of the properties of the biomasses. The study not only provided thermal degradation characteristics of biomass in different atmospheres but also insight into the design and performance of pretreating biomass with lower operating cost.

2. Experimental

2.1. Reaction system

A schematic of the experimental system is shown in Fig. 1. The system was made up of a gas supply unit, a reactor, a product gas treatment unit and a cooling unit. The volumetric flow rates of air and nitrogen were individually controlled by two mass flow controllers (Alicat Scientific). The reactor comprised a cylindrical chamber and a heater (or electrical furnace). The diameter and the height of the chamber were 125 and 440 mm, respectively, and it was heated by the heater located outside the chamber. The sample was placed in the chamber for torrefaction or low-temperature carbonization and the heater was utilized to elevate and sustain the reaction temperature. The reaction temperature was controlled by a proportional integral derivative (PID) temperature controller, and the power of the heater was controlled by a solid state relay (SSR) power controller. In the product gas treatment unit, pure water in two conical flasks in series was employed to remove tars and clean the exhaust gas. After the sample was torrefied, it was moved into the cooling unit. In the unit, the sample was placed in a vessel in which nitrogen with a flow rate of 1000 mL min^{-1} at 25 °C was used to directly cool the sample to room temperature. Meanwhile, the vessel was enveloped by a water jacket. Cooling water with a flow rate of 1000 mL min^{-1} at 10 °C was used to cool the vessel, thereby indirectly cooling the sample.

Table 1
Fiber, proximate, elemental and calorific analyses of raw oil palm fiber and eucalyptus.

Raw material	Oil palm fiber	Eucalyptus
Fiber analysis (wt%)		
Hemicellulose	44.2	18.1
Cellulose	34.9	56.9
Lignin	20.9	25.0
Proximate analysis (wt%, db[a])		
Volatile matter (VM)	72.5	81.1
Fixed carbon (FC)	20.5	18.9
Ash	7.0	0.0
Elemental analysis (wt%, db)		
C	51.9	50.8
H	4.8	5.1
N	2.4	0.0
O (by difference) [b]	40.6	44.1
S	0.3	0.0
HHV (MJ kg^{-1})	17.1	18.4

[a] Dry basis.
[b] O (wt%) = 100–C–H–N–S.

2.2. Experimental procedure

Oil palm fibers (OPF) was trimmed and meshed to a length less than 30 mm and needle-shaped pieces were obtained. Eucalyptus was cut into blocks with the dimensions of 10 × 15 × 8 mm. The biomass samples were dried in an oven at 105 °C. The moisture content was measured by a moisture analyzer (Ohaus MB45). In the pretreating experiments, the weights of OPF and eucalyptus were 10 g (±5%) and 20 g (±5%), respectively, as a consequence of different densities. The samples in the reactor were heated at a heating rate of 30 °C min^{-1} until the desired temperature was reached. Thereafter, they were torrefied/low-temperature carbonized for 1 h at flow rate of carrier gas (nitrogen or air) at 1000 mL min^{-1} (25 °C). The carrier gas was continuously blown into the reaction chamber to keep the system in an inert/oxidative environment and remove volatiles produced in the reactor. After the gas mixture left the reactor, they were cooled and washed in the conical flasks. The solid residues were cooled to room temperature in the cooler for further analysis.

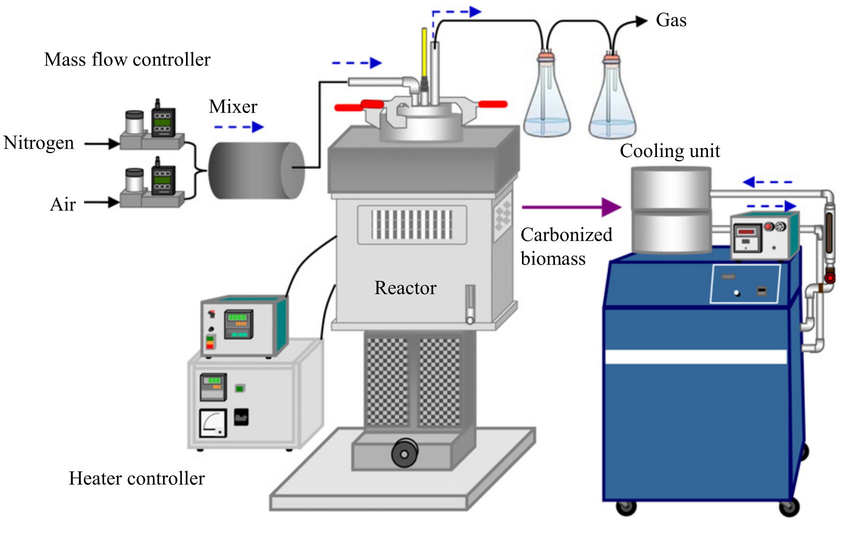


Fig. 1. Schematic presentation of experimental setup.

Hemicellulose, cellulose and lignin were analyzed as described in a previous study (Chen et al., 2010). The proximate analysis was performed in accordance with the standard procedure of the American Society for Testing and Materials (ASTM E870-82). The elemental analysis was carried out using an elemental analyzer (Vario EL III) to detect the weight percentages of C, H, N and S in biomass. The calorific values of samples were detected by a bomb calorimeter (IKA C2000 Basic). Nitrogen at a controlled flow rate was blown into the system and the flow rate of the gas at the exit of the system was detected to ensure mass conservation, whereby no leakage occurred. Experiments were carried out more than three times. The data shown below were average values and the relative error was less than 5% between repeats.

3. Results and discussion

The results of the fiber, proximate and elemental analyses as well as higher heating value (HHV) are listed in Table 1. The hemicellulose content in OPF was higher than that in eucalyptus, which

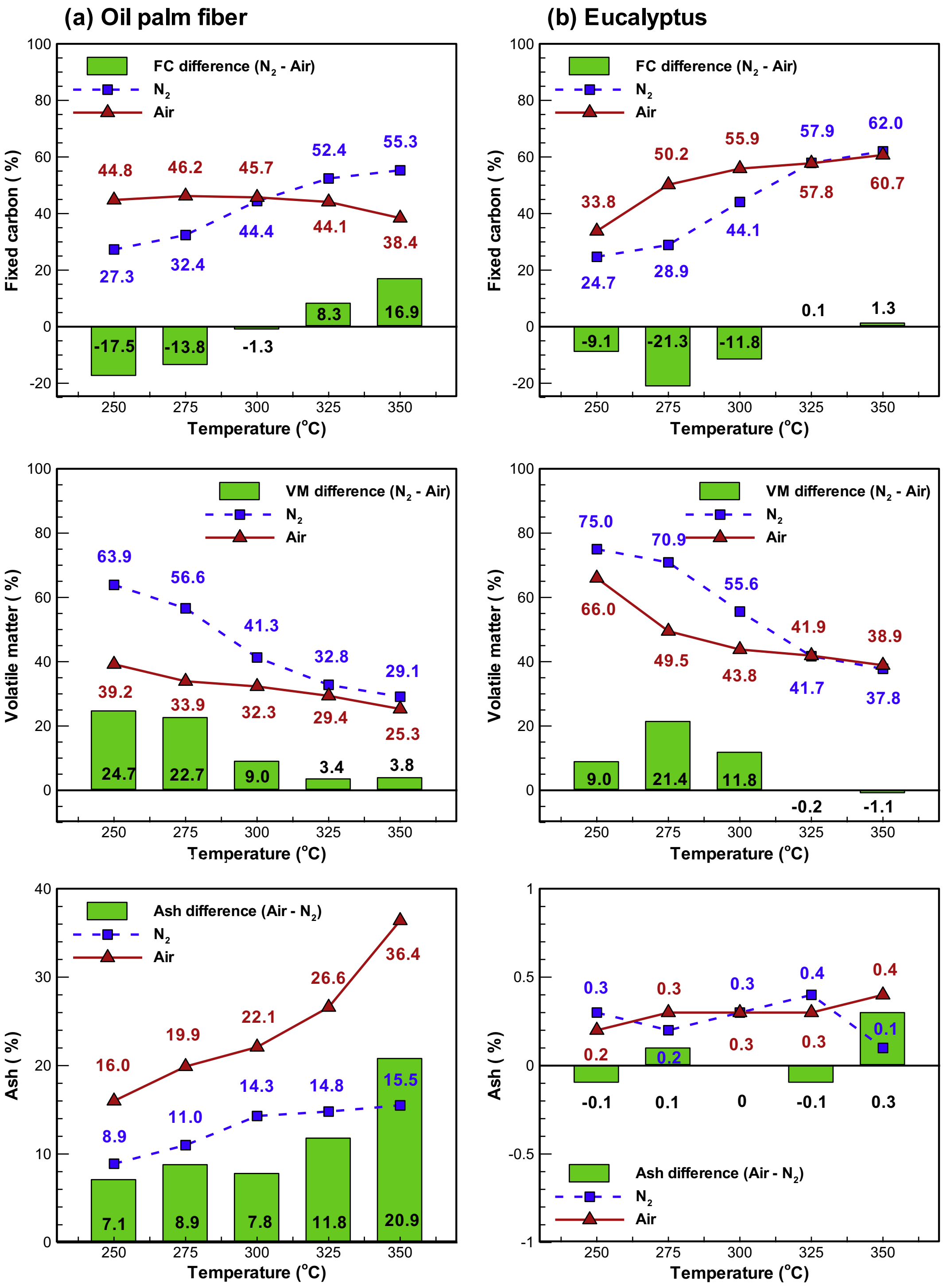


Fig. 2. Fixed carbon, volatile matter and ash contents of pretreated (a) oil palm fiber and (b) eucalyptus in N_2 and air atmospheres.

Table 2
Elemental analyses of raw and low-temperature carbonization biomasses.

Biomass	Gas	Elemental (wt%, db[a])	Temperature (°C)					
			Raw	250	275	300	325	350
Oil palm fiber								
	N_2	C	48.5	52.8	54.9	61.3	65.5	66.4
		H	4.4	5.1	5.0	4.3	3.4	3.3
		O[b]	37.5	31.7	27.5	18.5	14.2	12.7
		N	2.3	1.3	1.5	1.4	1.9	1.8
		S	0.3	0.2	0.1	0.2	0.2	0.1
		Ash	7.0	8.9	11.0	14.3	14.8	15.5
	Air	C		56.9	56.0	55.9	53.3	50.5
		H		2.9	2.3	2.1	1.8	2.0
		O[b]		21.7	19.0	16.9	14.7	6.8
		N		2.3	2.5	2.7	3.3	4.1
		S		0.2	0.2	0.2	0.3	0.2
		Ash		16.0	19.9	22.1	26.6	36.4
Eucalyptus								
	N_2	C	50.8	57.8	59.7	68.2	75.0	75.1
		H	5.1	4.9	4.7	4.2	3.6	3.6
		O[b]	44.1	37.0	35.4	27.3	21.0	21.1
		N	0.0	0.0	0.0	0.0	0.0	0.0
		S	0.0	0.0	0.0	0.0	0.0	0.0
		Ash	0.0	0.3	0.2	0.3	0.4	0.1
	Air	C		60.6	69.0	71.3	73.2	74.7
		H		4.2	3.0	2.2	1.5	1.2
		O[b]		35.0	27.7	26.3	25.0	23.7
		N		0.0	0.0	0.0	0.0	0.0
		S		0.0	0.0	0.0	0.0	0.0
		Ash		0.2	0.3	0.3	0.3	0.4

[a] Dry basis.
[b] By difference.

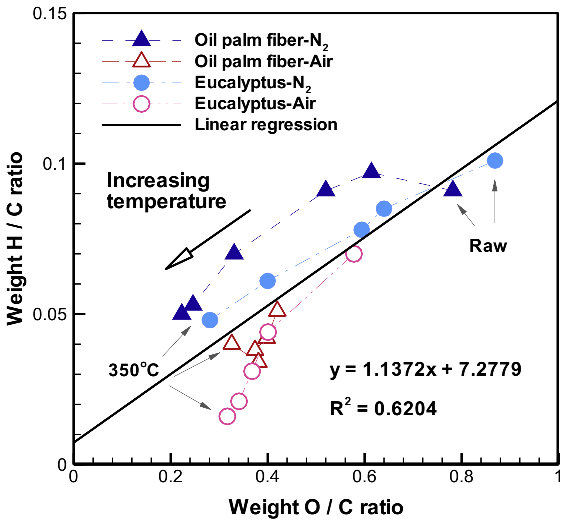
had a higher content of cellulose. Since Chen and Kuo (2010) found that the impact of torrefaction on biomass was low at torrefaction temperatures less than and equal to 240 °C, treatments were carried out at 250–350 °C for 1 h.

3.1. Proximate analysis

Higher heating value (HHV) is the most important property for biomass as a fuel and it is highly related to the proximate analysis (Parikh et al., 2005). In a N_2 atmosphere, the fixed carbon (FC) content of OPF monotonically increased with increasing temperature (Fig. 2a). In contrast, the maximum FC was exhibited at 275 °C for the biomass in air. When the temperature was lower than 300 °C, oxygen contained in air was conducive to the thermal degradation of OPF so that its FC in the air atmosphere was higher than in N_2. When the temperature was higher than 300 °C, the oxidation of OPF in air became pronounced, resulting in an opposite trend in FC. Overall, the difference in FC for OPF in N_2 and air increased with increasing temperature. An increase in temperature reduced volatile matter (VM) regardless of the atmosphere. At lower temperatures such as 250 and 275 °C, a large amount of VM was retained in OPF torrefied in N_2. Alternatively, for OPF pretreated in air at the same temperatures, VM was consumed markedly. For this reason, the difference in VM for OPF in N_2 and air at 250 °C was 24.7%. Once the temperature reached 350 °C, the difference diminished, implying that the consumption of VM was almost independent of the atmosphere at this specified temperature. For OPF in N_2, the ash content was between 8.9% and 15.5%, and it was from 16.0% to 36.4% in air. A higher value of ash content means less combustible components kept in the pretreated biomass. Since the difference in ash content for OPF in the two atmospheres at 350 °C was 20.9%, it is inadequate to pretreat OPF using air as a carrier gas.

The FC for eucalyptus in air at lower temperatures of 250–300 °C was always higher than that in N_2 (Fig. 2b), but the difference tended to become small at 325 and 350 °C (0.1% and 1.3%, respectively). A similar behavior was observed for VM. The most significant difference in FC and VM occurred at 275 °C, with values of 21.3% and 21.4%, respectively. Thus, the increase in FC for eucalyptus in air originated mainly from the depletion of VM. No ash was detected in eucalyptus (Table 1), implying that the ash content was extremely low in the biomass. Although the contents of volatile matter and fixed carbon were lowered by the thermal pretreatments in N_2 and air, the ash content remained low (0.1–0.4 wt%), as shown in Table 1.

(a) Weight ratio



(b) Atomic ratio

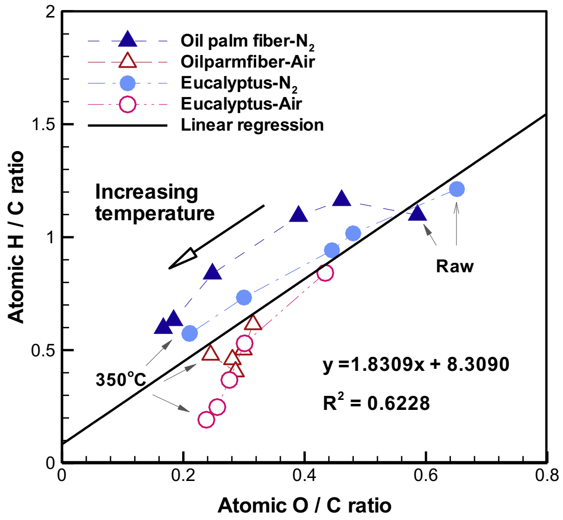


Fig. 3. H/C versus O/C ratio in terms of (a) weight basis and (b) atomic basis.

3.2. Elemental analysis

The elemental analyses of the raw and pretreated biomasses are tabulated in Table 2, and the profiles of weight and atomic H/C ratio versus O/C ratio (i.e. the van Krevelen diagrams) are demonstrated in Fig. 3a and b, respectively. The figures indicate that the H/C and O/C ratios declined with increasing temperatures when N_2 was used as carrier gas. This trend is consistent with the observations in other studies (Bridgeman et al., 2008; Chen et al., 2011b; Phanphanich and Mani, 2011; Rousset et al., 2011; Uemura et al.,

Table 3
Chemical formula of raw and low-temperature carbonization biomasses.

Temperature (°C)		Oil palm fiber	Eucalyptus
Raw		$CH_{0.091}O_{0.782}N_{0.047}S_{0.005}$	$CH_{0.101}O_{0.869}N_{0.000}S_{0.000}$
N_2	250	$CH_{0.097}O_{0.614}N_{0.025}S_{0.003}$	$CH_{0.085}O_{0.640}N_{0.000}S_{0.000}$
	275	$CH_{0.091}O_{0.520}N_{0.027}S_{0.002}$	$CH_{0.078}O_{0.594}N_{0.000}S_{0.000}$
	300	$CH_{0.070}O_{0.331}N_{0.024}S_{0.002}$	$CH_{0.061}O_{0.400}N_{0.000}S_{0.000}$
	325	$CH_{0.053}O_{0.246}N_{0.029}S_{0.002}$	$CH_{0.048}O_{0.281}N_{0.000}S_{0.000}$
	350	$CH_{0.050}O_{0.223}N_{0.027}S_{0.002}$	$CH_{0.048}O_{0.281}N_{0.000}S_{0.000}$
Air	250	$CH_{0.051}O_{0.420}N_{0.041}S_{0.004}$	$CH_{0.070}O_{0.578}N_{0.000}S_{0.000}$
	275	$CH_{0.042}O_{0.398}N_{0.045}S_{0.004}$	$CH_{0.044}O_{0.401}N_{0.000}S_{0.000}$
	300	$CH_{0.038}O_{0.374}N_{0.048}S_{0.004}$	$CH_{0.031}O_{0.368}N_{0.000}S_{0.000}$
	325	$CH_{0.034}O_{0.381}N_{0.062}S_{0.006}$	$CH_{0.021}O_{0.341}N_{0.000}S_{0.000}$
	350	$CH_{0.040}O_{0.326}N_{0.082}S_{0.004}$	$CH_{0.016}O_{0.317}N_{0.000}S_{0.000}$

2011; Wannapeera et al., 2011). In general, the weight and atomic H/C ratios of samples in N_2 are higher than those in air at the same temperatures. This can be explained by the intensified thermal degradation of the two biomasses in air so that the relative content of carbon in the samples is enriched (Table 2). When all the data are considered together, the slope of the regression line shown in Fig. 3b is equal to 1.83, implying that the impact of the thermal pretreatment on hydrogen is more than that on oxygen.

The chemical formulas of raw OPF and eucalyptus are $CH_{0.091}O_{0.782}N_{0.047}S_{0.005}$ and $CH_{0.101}O_{0.869}$, respectively (Table 3). That is, the O/C and H/C ratios in OPF are 0.782 and 0.091, respectively, and they are 0.869 and 0.101 in eucalyptus. After undergoing thermal pretreatment, the chemical formulas for OPF in N_2 and air at the temperature of 350 °C were transformed to $CH_{0.050}O_{0.223}N_{0.027}S_{0.002}$ and $CH_{0.040}O_{0.326}N_{0.082}S_{0.004}$, respectively, whereas they are $CH_{0.048}O_{0.281}$ and $CH_{0.016}O_{0.317}$ for eucalyptus in N_2 and air, respectively. In other words, for OPF pretreated by N_2 and air at 350 °C, the O/C and H/C ratios in the biomass became 0.223 and 0.050 as well as 0.326 and 0.040, respectively. It is obvious that hydrogen and oxygen contained in the biomasses were depleted to a certain extent from the pretreatment. The main components in non-condensable volatiles from the torrefaction of biomass were CO and CO_2, whereas the main components in condensable volatiles included H_2O, CH_3OH, HCOOH and CH_3COOH (Prins et al., 2006b; Deng et al., 2009). In the aforementioned gases or chemical components, the O/C and H/C ratios were much higher than those in the raw materials, implying that relatively more hydrogen and oxygen compared to carbon were released in the course of pretreatment. Hence the relative contents of H and O in the carbonized samples decreased.

3.3. Solid yield and higher heating value

Solid yields and yield differences of OPF and eucalyptus at various operating conditions are plotted in Fig. 4a, with the solid yield representing the weight ratio of residual solid to raw biomass. It has been reported that the thermal decomposition of hemicellulose occurred at the temperature range of 220 and 315 °C; cellulose decomposed mainly between 315 and 400 °C, and lignin was featured by gradual decomposition for the temperature ranging from

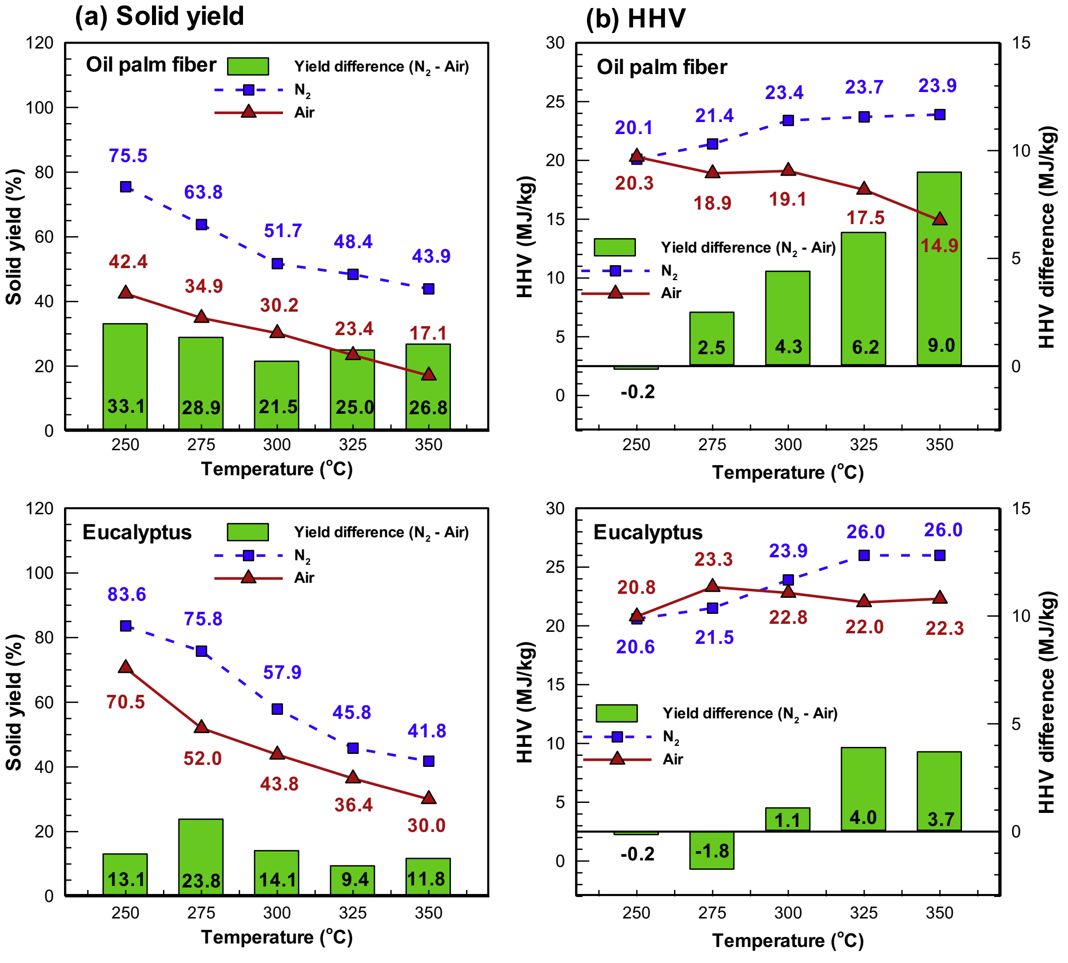


Fig. 4. (a) Solid yields and (b) higher heating values (HHV) of oil palm fiber and eucalyptus in N_2 and air atmospheres.

160 to 900 °C (Yang et al., 2007). Accordingly, the higher the temperature, the lower the solid yield. Past studies also showed that maximum decomposition rates of OPF and eucalyptus were achieved at around 280 (Luangkiattikhun et al., 2008; Idris et al., 2010) and 285 °C (Poletto et al., 2010), respectively. It follows that most of the hemicellulose was consumed in N_2 at the torrefaction temperature of 275 °C and a large portion of cellulose was depleted at 350 °C. For these reasons, the solid yields of the two biomasses in the N_2 atmosphere at 275 °C were substantially higher than those at 350 °C (Fig. 4a). The reactivity of cellulose and lignin depends on the nature of biomass (Chen and Kuo, 2010). At 300 or 350 °C, the solid yield of OPF under N_2 atmosphere was higher than that of eucalyptus. Because part of the cellulose and lignin will be consumed at the aforementioned temperatures, this reflects that cellulose and lignin in OPF may be less active than those in eucalyptus. In an oxidizing environment, the solid yield of OPF was lower than that of eucalyptus due to their different sizes (needle shape and block) and fibrous nature. The yield difference between OPF treated by N_2 and air atmospheres was larger than that of eucalyptus, possibly due to the higher hemicellulose content of OPF (= 44.2%) than of eucalyptus (18.1%), as shown in Table 1. In general, the yield difference for OPF in the two different atmospheres was more than 21%, whereas it was less than 14% for eucalyptus, except at 275 °C.

The HHVs of the two pretreated biomasses are presented in Fig. 4b. For OPF in N_2, the HHVs increased monotonically with increasing temperature, varying from 17.1 (raw) to 23.9 MJ kg^{-1} (350 °C). This observation is consistent with those from other studies (Bridgeman et al., 2008; Almeida et al., 2010; Pimchuai et al., 2010; Uemura et al., 2011; Wannapeera et al., 2011). In an air atmosphere, an increase in temperature resulted in a decrease in HHV of OPF. For instance, the HHV of OPF in air at 350 °C was 14.9 MJ kg^{-1}, a decrease of 17% in the calorific value of raw OPF. As a result, the difference of HHV between the N_2 and air atmospheres tended to grow as the temperature went up. Again, this finding reveals that using air as a carrier gas to pretreat OPF is not appropriate. When eucalyptus was pretreated in N_2 at 350 °C, its HHV increased from 18.4 to 26.0 MJ kg^{-1}, an increase of 41%. In air, the HHV was in the range of 20.8–23.3 MJ kg^{-1} and the maximum difference in HHV between the N_2 and air atmospheres was 4.0 MJ kg^{-1} (at 325 °C). This result suggests that in air the impact of pretreatment on eucalyptus was not as significant as that on OPF.

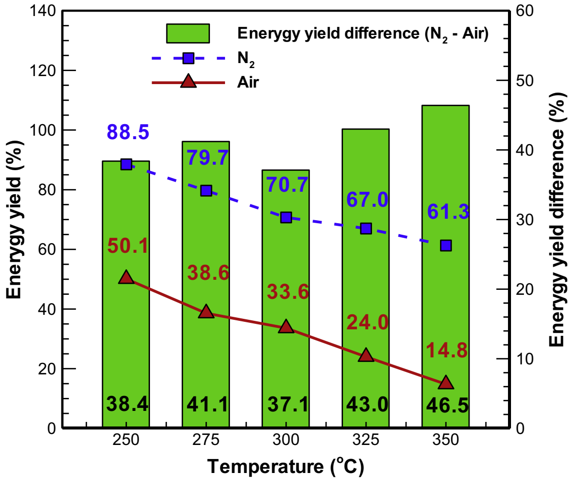
3.4. Energy yield

According to the solid yield and HHV (Fig. 4), the energy yields of OPF and eucalyptus at various operating conditions are displayed in Fig. 5. The energy yield was defined as

$$\text{Energy yield}(\%) = \frac{\text{HHV}_{\text{pretreated biomass}} \times \text{weight of pretreated biomass}}{\text{HHV}_{\text{raw biomass}} \times \text{weight of raw biomass}} \times 100 \qquad (1)$$

It can be seen that the higher the temperature, the lower the energy yield. Over 80% of the energy was retained when OPF was pretreated in N_2 at a temperature below 275 °C (Fig. 5a). In contrast, when OPF was pretreated in air at 250 °C, just 50% of energy yield was obtained. At 350 °C, the energy yield even dropped to 14.8% and the maximum difference of energy yield between the two different atmospheres was 46.5%. For eucalyptus in N_2, the energy yield was always larger than 59% within the investigated range of temperature. For biomass in air, once the temperature was higher than 300 °C, the energy yield fell below 50%. As a whole, the difference in energy yield between the two atmospheres was between 21.0% and 22.6%, except at 250 °C.

(a) Oil palm fiber



(b) Eucalyptus

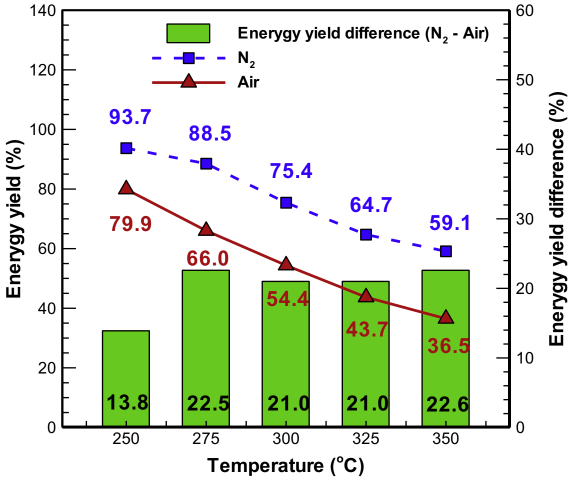


Fig. 5. Energy yields of (a) oil palm fiber and (b) eucalyptus in N_2 and air atmospheres.

3.5. Optimum operation

From solid fuel, it is desirable to obtain a high energy yield at a low solid volume from the pretreatment to increase processing efficiency and to facilitate transport. An energy-mass co-benefit index (EMCI) representing the difference between the energy yield and the solid yield, was defined to seek the optimum operation. The profiles of energy yield, solid yield and EMCI at various operating conditions are provided in Fig. 6. For OPF in N_2 and air, the maximum values of EMCI were located at 300 and 250 °C, respectively, implying that optimum operations occur at these temperatures. For eucalyptus in N_2 and air, the optimum operations take place at 325 and 275 °C, respectively. Since flue gases from combustion processes have temperatures around 350 °C (Kakaras et al., 2007), it might be possible to utilize flue gases as heat sources to pretreat biomass. The results of pretreatment with flue gases might lie between those using N_2 and air as carried gases because the oxygen contents in the flue gases are generally larger than zero but less than 21%.

4. Conclusions

Using N_2 as carrier gas, torrefaction of oil palm fiber and eucalyptus at 250–350 °C for 1 h provides desirable results, but only

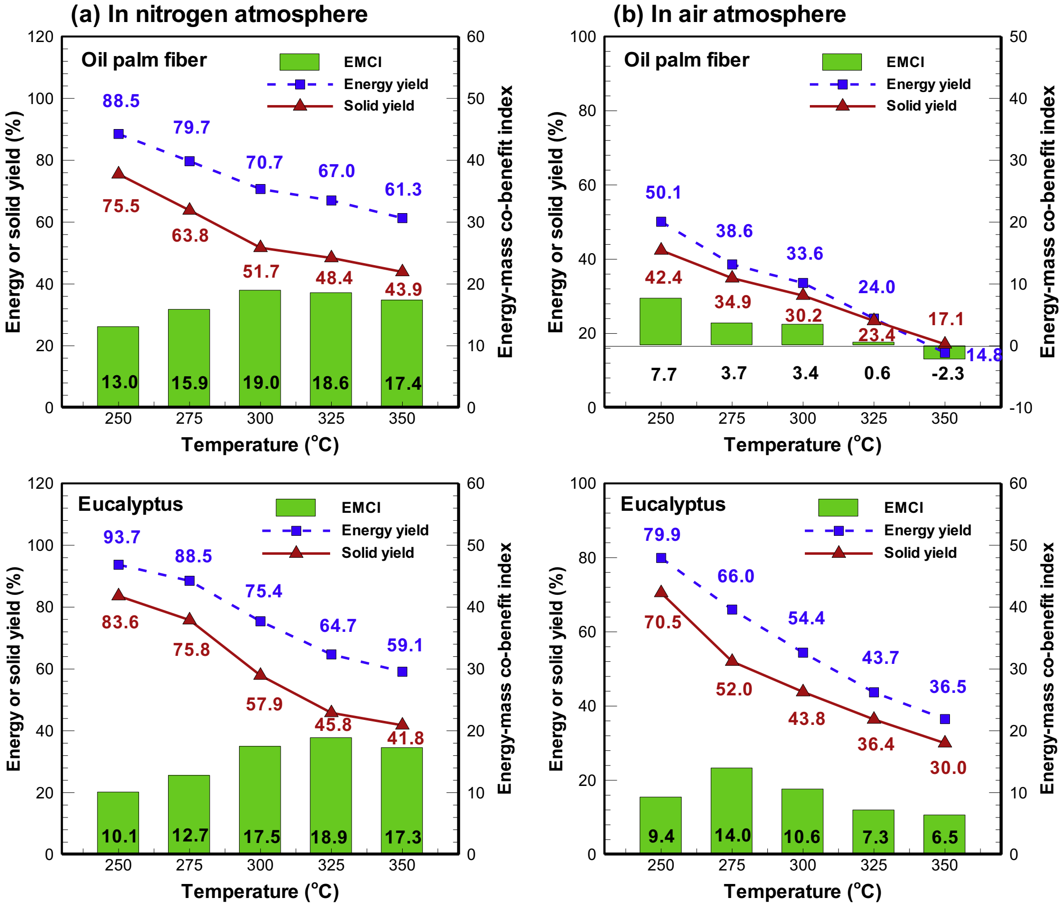


Fig. 6. Energy yields, solid yields and energy-mass co-benefit indexes (EMCI) of oil palm fiber and eucalyptus in (a) N_2 and (b) air atmospheres.

eucalyptus can be pretreated with air as a lower-cost carrier gas. Introduction of an energy-mass co-benefit index (EMCI) enables identification of optimal pretreatment conditions that satisfy the requirement of producing solid fuel with high energy density and low volume.

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